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(54) Title: IN-SITU BIO-OXIDATION OF LOW-GRADE REFRACTORY SULPHIDE MINERALS

(57) Abstract: A method for the *in-situ* bio-oxidation of low-grade refractory sulphide minerals, the method comprising the steps of: aerating an upper layer of a body of low-grade refractory sulphide minerals to a pre-determined depth; flooding the upper layer of the body with an acidic solution causing dissolution of metals therefrom, forming a pregnant solution; and collecting the pregnant solution from the body.

"In-Situ Bio-oxidation of Low-Grade Refractory Sulphide Minerals"**Field of the Invention**

The present invention relates to a method for the *in-situ* bio-oxidation of low-grade refractory sulphide minerals. More particularly, the present invention relates to a

- 5 method for the *in-situ* bio-oxidation of *low-grade* refractory sulphide minerals by microbial leaching.

Background Art

Bacterial leaching of low-grade refractory sulphide ores, is known. Typically, the low-grade refractory sulphide ore is finely divided and heaped, before an acidic

- 10 solution is sprayed thereon, encouraging indigenous chemolithotrophic bacterial activity. However, the applicability of conventional bacterial leaching methods is limited by the generally low reaction rates associated with such. Further, some low-grade refractory sulphide resources, such as gold extraction tailings, exhibit limited percolation properties, rendering conventional microbial leaching methods
15 largely ineffective.

It is one object of the present invention to provide a method for the *in-situ* bio-oxidation of refractory sulphide minerals by bacterial leaching exhibiting improved reaction rates over conventional methods. It is a further object of the present invention to provide a method for the *in-situ* bio-oxidation of refractory sulphide

- 20 minerals by bacterial leaching effective in the treatment of low-grade refractory sulphide ores exhibiting limited percolation.

The preceding discussion of the background to the invention is intended to facilitate an understanding of the present invention. However, it should be appreciated that the discussion is not an acknowledgement or admission that any

- 25 of the material referred to was part of the common general knowledge in Australia as at the priority date of the application.

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Throughout the specification, unless the context requires otherwise, the word "comprise" or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated integer or group of integers but not the exclusion of any other integer or group of integers.

5 Disclosure of the Invention

In accordance with the present invention, there is provided a method for the *in-situ* bio-oxidation of low-grade refractory sulphide minerals, the method comprising the steps of:

- 10 aerating an upper layer of a body of low-grade refractory sulphide minerals to a pre-determined depth;
- flooding the upper layer of the body with an acidic solution causing dissolution of metals therefrom, forming a pregnant solution; and
- collecting the pregnant solution from the body.

- 15 The body of low-grade refractory sulphide minerals may be provided in the form of a tailings dam, dump or heap.

Preferably, the method of the present invention further comprises the steps of:

- removing the upper layer from the body of low-grade refractory sulphide minerals to produce a fresh upper layer;
- 20 aerating the fresh upper layer of the body of low-grade refractory sulphide minerals to a pre-determined depth;
- flooding the fresh upper layer of the body of low-grade refractory sulphide minerals with an acidic solution causing dissolution of metals therefrom, forming a pregnant solution;
- collecting the pregnant solution from the body; and

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repeating the above steps as required.

Preferably, the method of the present invention further comprises the step of:

treating the removed upper layer to recover metals therefrom.

- The steps of flooding the upper layer of the body with an acidic solution and
5 collecting the pregnant solution may be repeated as required before an upper
layer is removed. In a preferred form of the invention, at least a portion of the
pregnant solution is used to generate the acidic solution.

Further and still preferably, the method of the present invention comprises the
step of:

- 10 monitoring metal levels in the upper layer of the body of low-grade
refractory sulphide minerals.

Preferably still, the method of the present invention further comprises the steps of:

monitoring metal levels in the pregnant solution from the body of low-grade
refractory sulphide minerals; and

- 15 passing at least a portion of pregnant solution to a metal-recovery circuit.

Preferably still, the method of the present invention comprises the additional step
of:

washing the removed upper layer and passing the washings therefrom to
the metal recovery circuit.

- 20 Preferably, after the step of aerating the or each upper layer of the body of low-
grade refractory sulphide minerals, the method comprises the step of:

promoting microbial activity in the or each upper layer.

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Preferably still, the step of promoting microbial activity in the or each upper layer is performed by inoculating the or each upper layer with an inoculum containing chemolithotrophic microbes.

In one form of the invention, the inoculum is formed by the steps of:

- 5 enriching naturally occurring chemolithotrophic microbes from the mineral heap;
 - selecting chemolithotrophic microbes adapted to function and grow under the conditions; and
 - cultivating the chemolithotrophic microbes to form the inoculum.
- 10 Preferably, the chemolithotrophic microbes are capable of oxidising sulphur and iron. In one specific form of the invention, the inoculum contains chemolithotrophic microbes of one or more of the following species: *Thiobacillus ferrooxidans*, *Thiobacillus thiooxidans* and *Leptospirillum ferrooxidans*. In an alternate form of the invention, the inoculum contains microbes of the *Sulfolobus* type.
- 15

Preferably, the microbial concentration of the inoculum is between about 10^6 and 10^{10} cells per mL.

Preferably, the acidic solution is of a pH between about 1.5 and 2.5.

- Conveniently, the inoculum is introduced with the acidic solution. In a specific form of the invention, the ratio of inoculum to acidic solution is approximately 1:1 by volume. Typically, the pH of the solution generated by adding the inoculum to the acidic solution is between about 1.5 and 2.5. In a highly specific form of the invention, the pH of the solution generated by adding the inoculum to the acidic solution is approximately 1.8.
- 25 Preferably, the or each step of aerating the surface layer comprises scarifying the surface layer.

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Preferably, the pre-determined depth is between about 200mm to about 500mm.

Best Mode(s) for Carrying Out the Invention

The best mode of performing the invention presently known to the applicant will now be described, by way of example only, with reference to one embodiment of 5 the invention, which relates to the recovery of gold and base metals from dammed gold tailings. However, it must be understood that the scope of the invention is not limited thereto.

In the embodiment, indigenous iron and sulphur oxidising microbes are identified 10 from the dam and cultivated to produce an inoculum rich in such, before an upper surface of the gold tailings dam is aerated by scarification to a depth of between 200mm and 500mm. The upper layer is then flooded with an acidic solution containing the inoculum. After a suitable time, the solution is drained or pumped 15 from the dam collection point and recirculated. Concentrations of base metals, such as copper and cobalt, in the run-off are monitored. When the base metal concentrations in the run-off reach a predetermined level, the solution is diverted 20 to a base metal extraction circuit. Further, the tailings solids are periodically tested for gold recovery, and when a predetermined level of oxidation and gold recovery is reached the upper layer is removed, thereby forming a fresh upper layer, and washed. The washings, together with the run-off, are passed to a metal recovery circuit, whilst the solid residue is passed to a gold recovery circuit.

The fresh upper layer is then aerated by scarification to a depth of between 200mm and 500mm, and the process repeated until the heap is treated.

Example

The efficacy of the present invention will now be illustrated by way of the following 25 example, in which data generated by a second embodiment of the invention is compared to data generated by a conventional *in-situ* biological oxidation technique.

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Gold extraction tailings (Peko Tailings from Tenant Creek) were treated by traditional biological heap leaching methods, involving preparing a pad and spraying acidified water onto the surface of the tailings. The dissolution of cobalt and copper was periodically tested, as shown in Table 1, below. Gold recoveries
 5 were not tested.

Treatment Period (days)	Cobalt Dissolution (%)	Copper Dissolution (%)
0	3	4
52	6	7
105	8	14
128	9	22
158	10	27
196	12	33
234	15	27
283	30	44
338	35	69
387	55	67

Table 1

Thus, using conventional biological heap leaching methods, 55% cobalt dissolution and 67% copper dissolution were recorded after 387 days.

Samples of the Peko gold extraction tailings were then treated in the laboratory.
 10 Samples of the gold extraction tailings were placed in watertight trays. An inoculum was generated by culturing the naturally occurring microbes from the samples. A portion of a sample was combined with water, and the water examined under an optical microscope. Microbes including *Thiobacillus ferrooxidans*, *Thiobacillus thiooxidans* and *Leptospirillum ferrooxidans* were
 15 identified. The indigenous microbes were cultivated by conventional means before being introduced into a vessel containing an acidic solution and low-grade sulphide ore, where concentrations of microbes were allowed to reach approximately 10^6 and 10^{10} cells per mL. Subsequent portions of inoculum were generated by simply refilling the vessel and allowing the microbes to proliferate.

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Each sample was aerated by scarification, and a solution of pH between 1.5 and 2.5 was combined with the inoculum to a ratio of 1:1 by volume, and used to flood an upper layer of the sample.

Cobalt, copper and gold dissolution after predetermined periods are reported in
 5 Table 2. Gold dissolution results were based on bottle roll cyanidation test work on the oxidation residues.

Treatment Time (days)	Gold Dissolution (%)	Cobalt Dissolution (%)	Copper Dissolution (%)	Sulphide Removal (%)
Untreated	18.3	3.2	0.14	0
55	41.5	41.7	57.7	33.8
95	89.5	44.8	61.3	50.2
110	91.7	49.1	64.3	51.2

Table 2

In addition to the effect of improving metal dissolution, application of the process resulted in a decrease in the consumption of cyanide and lime during the bottle
 10 roll cyanidation test work. The reagent consumptions are provided in Table 3.

Treatment Time (days)	Cyanide Consumption (kg/t)	Lime Consumption (kg/t)
Untreated	10	27
55	1.2	6.6
95	0.4	6.4
110	0.4	5.6

Table 3

As can be seen from the above comparison, the present invention provides a method for the *in-situ* bio-oxidation of refractory sulphide minerals by bacterial leaching exhibiting improved reaction rates over traditional biological heap
 15 leaching methods, involving preparing a pad and spraying acidified water onto the surface of the tailings. Further, the present invention provides a method for the *in-*

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situ bio-oxidation of refractory sulphide minerals by bacterial leaching effective in the treatment of low-grade refractory sulphide ores exhibiting limited percolation.

Modifications and variation such as would be apparent to the skilled addressee are considered to fall within the scope of the present invention.

The Claims Defining the Invention are as Follows

1. A method for the *in-situ* bio-oxidation of low-grade refractory sulphide minerals, the method characterised by the steps of:

aerating an upper layer of a body of low-grade refractory sulphide minerals
5 to a pre-determined depth;

flooding the upper layer of the body with an acidic solution causing dissolution of metals therefrom, forming a pregnant solution; and

collecting the pregnant solution from the body.

- 10
2. A method according to claim 1 characterised in that the body of low-grade refractory sulphide minerals is provided in the form of a tailings dam, dump or heap.
 3. A method according to claim 1 or claim 2 characterised by the further steps of:

removing the upper layer from the body of low-grade refractory sulphide minerals to produce a fresh upper layer;

15 aerating the fresh upper layer of the body of low-grade refractory sulphide minerals to a pre-determined depth;

flooding the fresh upper layer of the body of low-grade refractory sulphide minerals with an acidic solution causing dissolution of metals therefrom, forming a pregnant solution;

20 collecting the pregnant solution from the body; and

repeating the above steps as required.

4. A method according to claim 3 characterised by the steps of

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treating the removed upper layer to recover metals therefrom.

5. A method according to any one of the preceding claims characterised in that the steps of flooding the upper layer of the body with an acidic solution and collecting the pregnant solution may be repeated as required before an upper layer is removed.
6. A method according to any one of claims 3 to 5 characterised in that at least a portion of the pregnant solution is used to generate the acidic solution.
7. A method according to any one of the preceding claims characterised by the step of:
 - 10 monitoring metal levels in the upper layer of the body of low-grade refractory sulphide minerals.
 8. A method according to any one of the preceding claims characterised by the steps of:
 - 15 monitoring metal levels in the pregnant solution from the body of low-grade refractory sulphide minerals; and
 - passing at least a portion of pregnant solution to a metal-recovery circuit.
 9. A method according to any one of claims 3 to 8 characterised by the additional step of:
 - 20 washing the removed upper layer and passing the washings therefrom to the metal recovery circuit.
 10. A method according to any one of the preceding claims characterised by, after the step of aerating the or each upper layer of the body of low-grade refractory sulphide minerals, the step of:
 - promoting microbial activity in the or each upper layer.

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11. A method according to claim 10 characterised in that the step of promoting microbial activity in the or each upper layer is performed by inoculating the or each upper layer with an inoculum containing chemolithotrophic microbes.

12. A method according to claim 11 characterised in that the inoculum is formed
5 by the steps of:

enriching naturally occurring chemolithotrophic microbes from the mineral heap;

selecting chemolithotrophic microbes adapted to function and grow under the conditions; and

10 cultivating the chemolithotrophic microbes to form the inoculum.

13. A method according to claim 11 or 12 characterised in that the chemolithotrophic microbes are capable of oxidising sulphur and iron.

14. A method according to any one of claims 11 to 13 characterised in that the inoculum contains chemolithotrophic microbes of one or more of the following species: *Thiobacillus ferrooxidans*, *Thiobacillus thiooxidans* and *Leptospirillum ferrooxidans*.

15. A method according to any one of claims 11 to 13 characterised in that the inoculum contains chemolithotrophic microbes of the *Sulfolobus* type.

16. A method according to any one of claims 11 to 15 characterised in that the microbial concentration of the inoculum is between about 10^6 and 10^{10} cells per mL.

17. A method according to any one of the preceding claims wherein the acidic solution is of a pH between about 1.5 and 2.5.

18. A method according to any one of claims 11 to 17 characterised in that the inoculum is introduced with the acidic solution.
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19. A method according to claim 18 characterised in that the ratio of inoculum to acidic solution is approximately 1:1 by volume.
20. A method according to claim 17 or 18 characterised in that the pH of the solution generated by adding the inoculum to the acidic solution is between 5 about 1.5 and 2.5.
21. A method according to claim 20 characterised in that the pH of the solution generated by adding the inoculum to the acidic solution is approximately 1.8.
22. A method according to any one of the preceding claims characterised in that the or each step of aerating the surface layer comprises scarifying the surface 10 layer.
23. A method according to any one of the preceding claims characterised in that the pre-determined depth is between about 200mm to about 500mm.
24. A method substantially as described herein with reference to the first or second embodiment.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU02/00082

A. CLASSIFICATION OF SUBJECT MATTER		
Int. Cl. ⁷ : C22B 3/18, 3/00		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) IPC: C22B 3/18, 3/00		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) DWPI: SULPHID+, SULFID+, (BIO(W)OXID+), BIOXID+, BIOOXID+, TAILING+, HEAP+, DAM+, DUMP+, (IN(W)SITU), INSTITU		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 01/16385 A (BILLITON SA LIMITED) 8 March 2001 page 4, line 21 to page 6, line 29 and figure	1-24
A	WO 01/44519 A [PACIFIC ORE TECHNOLOGY (AUSTRALIA) LTD] 21 June 2001 whole document	1-24
A	WO 00/71763 A [BACTECH (AUSTRALIA) PTY LTD.] 30 November 2000 whole document	1-24
A	WO 00/37690 A (THE UNIVERSITY OF BRITISH COLUMBIA) 29 June 2000 whole document	1-24
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C <input checked="" type="checkbox"/> See patent family annex		
* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "B" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed		"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family
Date of the actual completion of the international search <u>1 March 2002</u>		Date of mailing of the international search report <u>15 MAR 2002</u>
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au Facsimile No. (02) 6285 3929		Authorized officer B. PREMARATNE Telephone No : (02) 6283 2407

INTERNATIONAL SEARCH REPORT

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5779762 A (KOHR et al.) 14 July 1998 whole document	1-24

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/AU02/00082

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report			Patent Family Member				
WO	200116385	AU	200073916				
WO	200144519	AU	200115061				
WO	200071763	AU	200043863				
WO	200037690	AU	200030684				
US	5779762	AU	39603/95	BR	9509472	CA	2203258
		GB	2308589	NZ	295938	WO	9612826
		ZA	9509037	US	5688304	US	6086656
END OF ANNEX							